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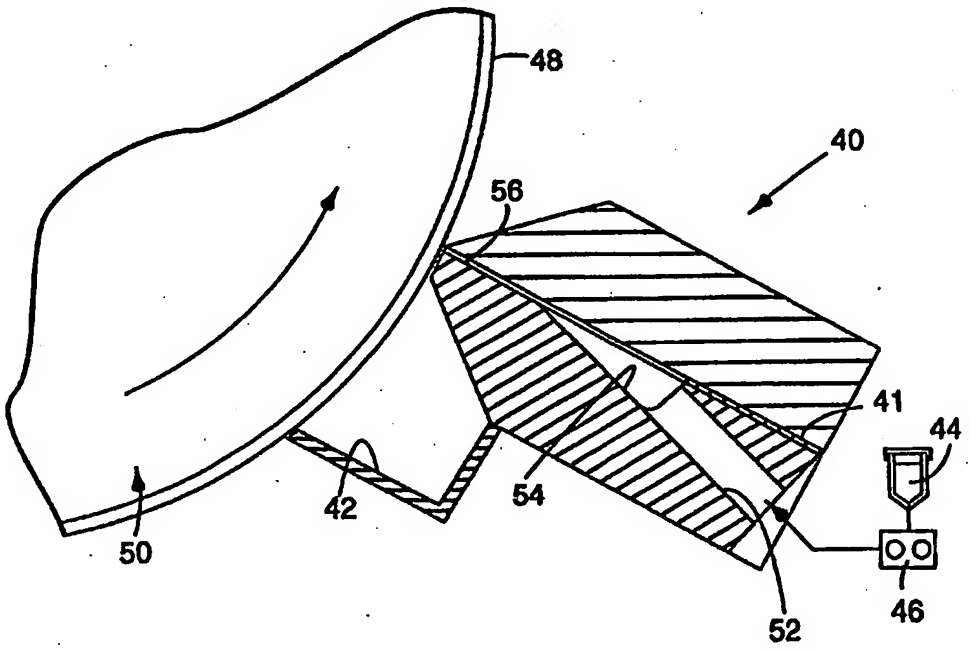

(57) Abstract

A method of coating the surface of a substrate with an essentially solvent-free polymerizable fluid that includes passing the fluid through a die onto the surface of the substrate as the substrate moves relative to the die. The die includes a channel adapted to receive the fluid and an adjustable width slot in communication with said channel through which the fluid is passed formed between a substantially straight, sharp edge located on the downstream side of the substrate and a land located on the upstream side of the substrate.

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PRECISION COATING PROCESS FOR PREPARING POLYMERIZABLE FILMS

Background of the Invention

Field of the Invention

This invention relates to a coating process for preparing polymerizable films.

Description of the Related Art

The bead coating method of applying fluids to substrates is known. According to this method, coating fluid is fed via a metering pump to a die which deposits the coating fluid on the surface of a moving substrate as the substrate moves past the die. As the coating fluid leaves the die it forms a continuous coating bead between the upstream die lip, the downstream die lip, and the web. The moving substrate is wetted by the bead as the substrate moves past the bead to create a layer of coating fluid on the substrate. To improve the stability of the bead (and thus reduce coating inhomogeneities), a vacuum may be applied to a vacuum chamber located upstream of the coating bead.

Summary of the Invention

In general, the invention features a method of coating the surface of a substrate with an essentially solvent-free (i.e., 100% solids) polymerizable fluid by passing the fluid through a die onto the surface of the substrate as the substrate moves relative to the die.

The die includes a channel adapted to receive the fluid and an adjustable width slot in communication with the channel through which the fluid is passed. The slot is formed between the downstream bar 66 and the upstream bar 64. The downstream bar lip is formed as a sharp edge 70 and th upstr am bar lip is formed as a land 68 which substantially corresponds in shape

to th shap of the substrate in the immediate area of coating fluid application. As used herein, "upstream" and "downstream" are relative to the direction of the moving substrate.

In preferred embodiments, the edge radius of the sharp edge (as defined in Fig. 3) measures no more than about 10 microns, and more preferably ranges from about 2 to about 4 microns. The edge angle A_1 of the sharp edge (as defined in Fig. 3) preferably ranges from about 20° to about 75° and preferably is about 50 - 60°.

The convergence of the die C (as defined in Fig. 3) preferably ranges from about 0° to about 2.29°, more preferably from about 0° to about 1.5°.

The sharp edge and the land are preferably configured such that the sharp edge is displaced towards the surface of the substrate relative to the land. The degree of displacement is referred to as "overbite" O. Preferably, the overbite is no greater than about 0.64 mm.

The sharp edge is substantially straight. For example, along a distance of about 25 cm measured anywhere along the sharp edge, the straightness of the edge does not vary by more than about 2.5 microns, and preferably no more than about 1 micron.

The rate at which the fluid passes through the die and the rate at which the substrate moves relative to the die are adjusted to provide a substantially uniform caliper coating on the substrate.

The viscosity of the coating fluid is preferably at least about 10 cps, and may be 100 cps or greater, or even 1000 cps or greater. The method may be adapted to apply both thin and thick coatings. During coating, a vacuum may be applied to the upstream side of the die to improv coating quality if desir d. The substrate may be a w b.

The invention enables the preparation of solvent-free coatings having uniform caliper in both the downweb and crossweb directions. Both thick and thin films can be prepared. The invention is useful in a variety of settings, including the preparation of optical quality thin films and adhesive films.

Other features and advantages of the invention will be apparent from the following description of the preferred embodiments thereof, and from the claims.

Brief Description of the Drawings

The invention will be more fully understood with reference to the following drawings in which:

Figure 1 is a cross-sectional view of an extrusion die of the present invention.

Figure 2 is an enlarged cross-sectional view of the slot and lip of the die of Figure 1.

Figure 3 is a cross-sectional view of the slot and lip similar to that of Figure 2.

Figure 4 is a cross-sectional view of an alternative vacuum chamber arrangement.

Figure 5 is a cross-sectional view of another alternative vacuum chamber arrangement.

Figure 6 is a cross-sectional view of an alternative extrusion die of the present invention.

Figures 7A and 7B are enlarged cross-sectional views of the slot, face, and vacuum chamber of the die of Figure 6.

Figures 8A and 8B are schematic views of the die of Figure 6.

Description of the Preferred Embodiments

This invention is a die coating method for coating polymerizable fluids onto substrates (e.g., webs) the die includes an upstream die lip formed as a sharp edge and a downstream die lip formed as a land. The shape f th land substantially corresponds to the shape of the substrat in the imm diate area of coating fluid

application. The shape of the substrate may be flat or curved.

Figure 1 shows an extrusion die 40 with a vacuum chamber 42 useful in the coating method according to the present invention. Polymerizable fluid 44 is supplied by a pump 46 to the die 40 for application to a moving substrate 48, supported by a backup roll 50. Polymerizable fluid 44 is supplied through a channel 52 to a manifold 54 for distribution through a slot 56 and coating onto the moving substrate 48. The height and width of slot 56 can be controlled by means of a U-shaped shim 41. The shim is typically made of brass or stainless steel.

As shown in Figure 2, the polymerizable fluid 44 passes through the slot 56 and forms a continuous coating bead 58 between the downstream edge 72 of land 68, the lip of downstream bar 66, and the substrate 48. Vacuum chamber 42 (Figure 1) applies vacuum upstream of the bead to stabilize the coating bead. If desired, the temperature of both die 40 and backup roll 50 may be controlled to improve coating rheology.

The polymerizable fluid can be one of numerous compositions. Polymerization may be thermally induced or radiation induced (e.g., ultraviolet radiation or electron beam). Examples of suitable polymerizable fluids include epoxies, acrylates, methacrylates, vinyl ethers, isocyanates, and mixtures thereof. The resulting coatings are useful in a variety of applications, including adhesives, optical quality films (e.g., polymer dispersed liquid crystal or "PDLC" films and optical adhesives), precision caliper films, and vibration damping materials. The coatings are particularly useful in applications requiring thin films with uniform caliper control.

The lip of the upstream bar 64 is formed as a curved land 68 and the lip of the downstream bar 66 is

formed as a sharp edge 70. Sharp edge 70 should be clean and free of nicks and burrs, and should be straight within 1 micron in 25 cm of length measured anywhere along the edge. The edge radius should be no greater than 10 microns. The radius of the curved land 68 should be equal to the radius of the backup roll 50 plus a minimal, and non-critical, 0.13 mm allowance for coating gap and substrate thickness.

Figure 3 shows dimensions of geometric operating parameters for single layer extrusion. The length L_1 of the curved land 68 on the upstream bar 64 can range from 1.6 mm to 25.4 mm. The preferred length L_1 is 12.7 mm. The edge angle A_1 of the downstream bar 66 can range from 20° to 75°, and is preferably 50-60°. The die attack angle A_2 between the downstream bar 66 surface of the coating slot 56 and the tangent plane P through a line on the substrate 48 surface parallel to, and directly opposite, the sharp edge 70 can range from 60° to 120° and is preferably 90° to 95°. The coating gap G_1 is the distance between the sharp edge 70 and the substrate 48.

Slot height H is the distance between upstream bar 64 and downstream bar 66, and is controlled by controlling the thickness of shim 41 (shown in Figure 1). In general, the slot height ranges from 0.076 mm to 1.27 mm.

Overbite O is a positioning of the sharp edge 70 of the downstream bar 66, with respect to the downstream edge 72 of the curved land 68 on the upstream bar 64, in a direction toward the substrate 48. Overbite also can be viewed as a retraction of the downstream edge 72 of the curved land 68 away from the substrate 48, with respect to the sharp edge 70, for any given coating gap G_1. Overbite can range from 0 mm to 0.64 mm, and the settings at opposite ends of the die slot should be within 2.5 microns of each other.

Conv rgence C is a count rclockwise, as shown in Figure 3, positioning of the curved land 68 away from a location parallel to the substrate 48, with the downstream edge 72 being the center of rotation. Convergence can range from 0° to 2.29°, and the settings at opposite ends of the die slot should be within 0.023° of each other.

Overbite, slot height and convergence together affect the ability of the coating die to hold a steady bead. The interaction between these variables depends upon the rheology of the polymerizable coating; accordingly, these variables, along with the substrate speed, are adjusted based upon the particular polymerizable coating being used.

Optimum coating quality is achieved when the die coating apparatus is isolated from ambient sources of vibration and/or other disrupting factors.

The vacuum chamber 42, as shown in Figure 4, can be an integral part of, or clamped securely to, the upstream bar 64 to allow precise, repeatable vacuum system gas flow. The vacuum chamber 42 is formed using a vacuum bar 74 and can be connected through a vacuum restrictor 76 and a vacuum manifold 78 to a vacuum source channel 80. As shown in Figure 4, a curved vacuum land 82 is attached directly to the upstream bar 64. The vacuum land 82 has the same radius of curvature as the curved land 68. The curved land 68 and the vacuum land 82 can be finish-ground together so they are "in line" with each other. The vacuum land 82 and the curved land 68 then have the same convergence with respect to the substrate 48.

The vacuum land gap G_2 is the distance between the vacuum land 82 and the substrate 48, and is the sum total of the coating gap G_1, the overbite, and the displacement caused by the convergence C of the curved land. When the vacuum land gap G_2 is large, an

excessive inrush of ambient air to the vacuum chamber 42 occurs. Even though the vacuum source may have sufficient capacity to compensate and maintain the specified vacuum pressure level at the vacuum chamber 42, the inrush of air can have undesirable effects.

In Figure 5, the vacuum land 82 is part of a vacuum bar 74 which is attached to the upstream bar 64. During fabrication, the curved land 68 is finished with the convergence "ground in." The vacuum bar 74 is then attached and the vacuum land 82 is finish ground, using a different grind center, such that the vacuum land 82 is parallel to the substrate 48, and the vacuum land gap G_2 is equal to the coating gap G_1 for one preselected value of the overbite. The vacuum land length L_2 may range from 6.35 mm to 25.4 mm. The preferred length L_2 is 12.7 mm. This embodiment has greater overall coating capability in difficult coating situations compared to the embodiment of Figure 4, but it is always finish ground for one specific set of operating conditions. Consequently, as coating gap G_1 or overbite O are changed vacuum land gap G_2 may move away from its optimum value.

In Figures 6, 7A, and 7B, the die 40 is mounted on an upstream bar positioner 84, and the vacuum bar 74 is mounted on a vacuum bar positioner 86. The curved land 68 on the upstream bar 64 and the vacuum land 82 on the vacuum bar 74 are not connected directly to each other. The vacuum chamber 42 is connected to its vacuum source through the vacuum bar 74 and the positioner 86. The mounting and positioning for the vacuum bar 74 are separate from those for the upstream bar 64. A flexible vacuum seal strip 88 seals between the upstream bar 64 and the vacuum bar 74.

The gap G_2 between the vacuum land 82 and the substrate 48 is not affected by coating gap G_1, overbite, or converg nce changes, and may be held at

its optimum value continuously, during coating. The vacuum land gap G_2 may be set within the range from 0.076 mm to 0.508 mm. The preferred value for the gap G_2 is 0.15 mm. The preferred angular position for the vacuum land is parallel to the substrate 48.

Figures 8A and 8B show some positioning adjustments and the vacuum chamber closure. Overbite adjustment OA translates the downstream bar 66 with respect to the upstream bar 64 such that the sharp edge 70 moves toward or away from the substrate 48 with respect to the downstream edge 72 of the curved land 68. Convergence adjustment CA rotates the upstream bar 64 and the downstream bar 66 together around an axis running through the downstream edge 72, such that the curved land 68 moves counterclockwise from the position shown in Figures 8A and 8B, away from parallel to the substrate 48, or clockwise back toward parallel. Coating gap adjustment CGA translates the upstream bar 64 and the downstream bar 66 together to change the distance between the sharp edge 70 and the substrate 48, while the vacuum bar remains stationary on its mount 86, and the vacuum seal strip 88 flexes to prevent air leakage during adjustments. Air leakage at the ends of the die into the vacuum chamber 42 is minimized by end plates 90 attached to the ends of the vacuum bar 74 which overlap the ends of the upstream bar 64. The vacuum bar 74 is 0.10 mm to 0.15 mm longer than the upstream bar 64, so, in a centered condition, the clearance between each end plate 90 and the upstream bar 64 will range from 0.050 mm to 0.075 mm.

The width of the coating produced by a given die is reduced where indicated by "deckling" the die and the vacuum chamber by concurrently incorporating a) shaped plugs to reduce the widths of the die cavity manifold 54 and vacuum chamber 42 to the deckling width

and b) a shim into the die that has a shim slot width corresponding to the deckling width.

During coating, it has been found that, as a consequence of the structure of die 40, bead 58 does not move down to any appreciable extent into the space between curved land 68 and the moving substrate 48, even as vacuum is increased. This allows the use of relatively high vacuum levels. Moreover, good results are obtained even in the absence of vacuum. In addition, the effect of "runout" in back-up roll 50 on downweb coating weight is minimized.

The above-described die structure coupled with careful control of (a) the rate at which the polymerizable composition is delivered to the die (through control of pump speed) and (b) the substrate speed results in coatings having uniform caliper in both the downweb and crossweb directions.

For applications where optical appearance of the article is critical, contamination resulting from airborne particulates can be reduced by coating substrates in a clean room environment.

The invention will now be more fully understood with reference to the following examples which are not to be construed as limiting the scope of the invention.

EXAMPLES

Test Procedure A

The electro-optical responses of the PDLC devices were characterized using a computer-controlled test stand consisting of an IBM personal computer interfaced with Kepco 125-1KVA-3T power supply, a Dyn-Optics Optical Monitor 590, and a Valhalla Scientific 2300 Series Digital Power Analyzer. The optics of the Dyn-Optics Optical Monitor were adjusted such that the specular transmission of photopically-filter light at an approximate 6° coll ction half angle was measured relative to an open beam.

A sampl of a PDLC film/electrode sandwich measuring several square centimeters was attached to the leads of the power supply using a connector such as that described in the aforementioned Engfer et al. application. A 60 Hz voltage ranging from zero to 120 volts AC (VAC) was applied to the sample in 5 VAC increments and the specular transmission recorded.

Test Procedure B

The haze of the powered (120 VAC, 60 Hz) PDLC devices was measured using a Pacific Scientific Gardner XL-835 Colorimeter according to the manufacturer's instructions.

Examples 1-6

A series of adhesives were prepared from prepolymer syrups consisting of a mixture of 90 wt.% isooctyl acrylate and 10 wt.% acrylic acid (Aldrich, Milwaukee, WI) containing 0.04 wt.% photoinitiator 2-phenyl-2,2-dimethoxy acetophenone (KB-1, Sartomer, West Chester, PA) as described in U.S. Pat. No. 4,330,590 (Vesley), which is incorporated herein by reference. The syrups were partially photopolymerized to viscosities of 360, 1950 and 5600 cps (as measured on a Brookfield viscometer using a #4 spindle operating at 60 rpm) by varying the exposure times.

After the syrups had been advanced to the indicated viscosities, an additional 0.1 wt.% KB-1 photoinitiator and 0.2 wt.% hexanediol diacrylate (Sartomer, West Chester, PA) were added to the syrups and the mixtures agitated until homogeneous fluids were obtained. The resulting fluids were coated on the substrates at the thicknesses indicated in Table 1 using a precision coating die as described above and the lamination apparatus described in Vesley et al., PCT International application No. _____ (Attorney's Docket No. 50777PCT7A) entitled "Lamination Process for Coatings," filed concurrently with th

present application and assigned to the same assign as the present application.

During the coating operation, the first substrate was unwound from a first unwind roll and passed over a free-wheeling, unheated steel backup roll 25.4 cm (10 inches) in diameter where a 10.2 cm (4 inch) wide strip of the prepolymer syrup, which was delivered to the precision coating die using a precision gear pump (available from Zenith Corp.), was coated onto the first surface of the first substrate using a 10.2 cm (4 inch) die with no vacuum applied to the vacuum chamber. In Examples 1-4, a coating die similar to that illustrated in Figure 4 was configured with a 0.50 mm (20 mil) shim, a 0° convergence, an overbite of 0.076 mm (3 mil), a coating land L_1 of 12.7 mm, a vacuum land L_2 of 12.7 mm, and a die attack angle A_2 of 90°. In Examples 5-6, a 20.3 cm (8 inch) wide strip of the prepolymer syrup was coated onto the first surface of the first substrate using a 20.3 cm (8 inch) die similar to that used for Examples 1-4 except that it was configured with a 0.048 mm (19 mil) shim and an overbite of 0.254 mm (10 mil). The coating gap was adjusted as indicated in Table 1 along with the pump speed and substrate speed to produce coatings having the indicated thicknesses. No vacuum was applied to the vacuum chamber during the coating operation.

The second substrate was unwound from a second unwind roll and passed around a 2.54 cm (1 inch) diameter sintered metal laminator bar where it was laminated to the coated face of the first substrate according to the procedure described in the aforementioned Vesley et al. application. The laminator bar was located approximately 12 cm (4.7 inches) downstream from the backup roll such that the c ated substrate was not in contact with the backup roll or other idler r takeup roll at the point of

lamination, and positioned so that the uncoated first substrate was depressed approximately 3.8 mm (150 mils) below the plane defined by the first substrate as it passed between the backup roll and the idler roll; the extent of depression is hereinafter referred to as "interference." Air pressure (approximately 2.1 bar) through the sintered metal bar was adjusted to provide a cushion of air between the laminator bar and the second substrate.

The thus produced uncured laminate construction was cured to a high performance pressure sensitive adhesive by passing the construction under a bank of fluorescent black lights lamps (F20T12-350BL, available from Osram Sylvania, Danvers, MA). The laminate construction was exposed to 360 mJ/cm^2 of irradiation as measured with a UVIRAD radiometer (model number UR365CH3, available from Electronic Instrumentation and Technology, Inc., Sterling, VA) equipped with a glass filter responsive between 300 and 400 nm, with a maximum transmission at 365 nm. The average light intensity in the curing zone was about 2.3 mW/cm^2. Coating speeds were controlled by a vacuum pull roll positioned at the end of the coating line and were maintained at approximately 5.5 m/min. (11 feet/min).

Table 1 shows typical coating variations for various coating thicknesses and viscosities. The cured adhesives of examples 5 and 6 adhered to the polyester when the laminated construction was peeled apart. Adhesive and shear properties of the cured polymer syrups of Examples 5-6 were consistent with the properties obtained from similar formulations cured under the conditions described in U.S. Pat. No. 4,330,590.

Table 1

Example	First Substrate	Second Substrate	Viscosity (cps)[1]	Coating Gap (mm)	Coating Thickness (mm)
1	PET[2]	PET[2]	365	0.175	0.223±0.004
2	PET[2]	PET[2]	365	0.175	0.154±0.003
3	PET[2]	PET[2]	1,950	0.175	0.116±0.001
4	PET[2]	PET[2]	1,950	0.175	0.221±0.001
5	Release Paper[3]	PET[2]	5,600	0.127	0.150±0.001
6	Release Paper[3]	PET[2]	5,600	0.05	0.93±0.08

1. Measured on a Brookfield viscometer using a #4 spindle operating at 60 rpm.
2. Biaxially oriented PET film, 51 microns (2 mils) thick.
3. Polyethylene-coated paper provided with a silicone release coating.

Example 7

A PDLC device was prepared from a fluid containing (a) 55 parts of a mixture consisting of 30.0 wt.% RCC-15C curable matrix mixture obtained without initiator and with 50% less thiol (W.R. Grace, Atlanta, GA), 7.5 wt.% acrylic acid, 30.0 wt.% isooctyl acrylate, 15.0 wt.% 2-phenoxyethyl acrylate (Sartomer, West Chester, PA), 15.0 wt.% divinyl ether of triethylene glycol (International Specialty Products, Wayne, NJ), and 2.5 wt.% KB-1 photoinitiator, and (b) 45 parts BL036 liquid crystal mixture (EM Industries, Hawthorne, NY) having a solution viscosity of 42 cps (measured on a Brookfield viscometer using a #3 spindle operating at 60 rpm). The fluid, which was degassed under vacuum for approximately 2 minutes at ambient temperature, was applied as a 15.2 cm (6 inch) wide strip to the electrode surface of an ITO-coated polyester film (90/10 indium/tin oxide ratio, 80 ohms/square, 51 microns (2 mil) thick PET, available from Southwall Technologies, Palo Alto, CA) at a rate of approximately

152.4 cm/min (5 ft/minute) using the precision coating process described in Examples 1-6 except that a 88.9 cm die similar to that illustrated in Figure 7a was used. This die was deckled to produce a narrower coating and configured with a 152 micron shim, a coating land having a length (L_1) of 12.7 mm, a vacuum land having a length L_2 of 12.7 mm, a 0.57° convergence, a 33 micron overbite, a vacuum land gap G_2 of 0.152 mm, a die attack angle A_2 of 95°, and a coating gap G_1 of 102 microns. The convergence of the vacuum bar was 0° and no vacuum was applied to the vacuum chamber during coating. Both the die and back-up roll were temperature controlled at 21°C. A pressure of 1.7 bar was maintained to the sintered metal bar during lamination and the lamination bar was adjusted to provide an interference of 3.6 mm.

The uncured laminate construction was cured by passing the construction through a cooled curing chamber constructed of ultraviolet transparent Acrylite™ OP-4 (available from Cyro Industries, Mt. Arlington, NJ), extending approximately 61 cm (2 feet) into a cure chamber equipped with two banks of fluorescent black lights (F20T12-350BL, available from Osram Sylvania, Danvers, MA), one bank positioned on each side of the laminate. Air temperature in the cooling chamber was monitored by a thermocouple mounted in the chamber under the second fluorescent bulb and controlled at the indicated temperature by introducing temperature controlled air. Each side of the laminate construction was exposed to approximately 530 mJ/cm^2 of radiation calculated from light intensities of 1.1 mW/cm^2 as measured through the conductive electrode used in the PDLC device by means of a UVIBRITE radiometer (model number UBM365MO, available from Electronic Instrumentation and Technology, Inc., Sterling, VA) quipped with a glass filter responsiv betw en 300 and 400 nm, with a maximum transmission at 365 nm. The

radiometer was specially calibrated to read in absolute intensity.

The backup roll 50 was a pacer roll driven by a Torquer Tachometer precision motor (available from Inland Motor Division, Bradford, VA).

The cured coating thickness of the resulting PDLC film was 24±1 microns. The PDLC device had on- and off-state transmissions of 73.1% and 1.2%, respectively, and a haze of 5.8%.

Example 8

A PDLC device was prepared as described in Example 7 except that the coating fluid had the following composition: (a) 50 parts of a mixture consisting of 20.0 wt.% Vectomer 2020 (Allied-Signal, Inc., Morristown, NJ), 5.0 wt.% acrylic acid, 25.0 wt.% isooctyl acrylate, 15.0 wt.% 2-phenoxyethyl acrylate, 10 wt.% trimethylolpropane tris(3-mercaptopropionate) (Aldrich, Milwaukee, WI), 22.5 wt.% cyclohexane dimethanol divinyl ether (International Specialty Products, Wayne, NJ) and 2.5 wt.% Escacure KB-1, and (b) 50 parts BL036 liquid crystal mixture. The viscosity of the coating fluid was 134 cps (measured on a Brookfield viscometer using a #3 spindle operating at 60 rpm). The coating temperature was 21°C and during lamination an air pressure of 2.4 bar was maintained to the laminator bar which was adjusted to provide an interference of 3.8 mm. The fluid was applied as a 15.2 cm (6 inch) wide strip to the electrode surface of an ITO-coated polyester film at a rate of approximately 152.4 cm/min (5 ft/minute) using the precision coating process described in Example 7 except that the die was configured with a 46 micron overbite, a coating gap of 102 microns, and a vacuum of 1.9 mm Hg (1 inch of water) was used to apply the solution at 22°C. The film was cured at 21°C by exposing each side to approximately

530 mJ/cm^2 at an intensity of 1.0 mW/cm^2 to produc a PDLC film with a thickness of 23±1 microns.

The PDLC device had on- and off-state transmissions of 71.9% and 1.1%, respectively, and a haze of 4.8%.

Example 9

A PDLC device was prepared as described in Example 7 except that the fluid contained 500 parts of BL036 liquid crystal mixture and 333 parts of a mixture having the composition of 2.5 wt.% Esacure KB-1 photoinitiator, 7.5 wt.% acrylic acid, 30.0 wt.% isooctyl acrylate, 15.0 wt.% 2-phenoxyethyl acrylate 15.0 wt.% Uralac 3004-102 (DSM Resins, U.S., Inc., Elgin, IL), and 30.0 wt.% Uralac 3004-300 (DSM Resins, U.S., Inc., Elgin, IL). The 88.9 cm wide die was configured with a slot width of 88.9 cm, an overbite of 43 microns, a vacuum land gap G_2 of 24.5 mm and a vacuum of 1.9 mm Hg was applied to the vacuum chamber during coating. The ITO-coated polyester film used for the electrodes was approximately 130 microns (5 mils) thick. An air pressure of 3.4 bar was maintained to the laminator bar which was adjusted to provide an interference of 6.35 mm. The resulting laminate was exposed UV light having an average intensity of approximately 1.68 mW/cm^2 at about 23°C to produce a PDLC film approximately 18 microns thick.

The PDLC device had on- and off-state transmissions of 73.4% and 1.7%, respectively, and a haze of 5.3%.

Example 10

A PDLC device was prepared as described in Example 7 except that a fluid containing (a) 57.5 parts of a mixture consisting of 13.7 wt.% lauryl methacrylate (Rhom Tech, Inc., Malden, MA), 3.9 wt.% methacrylic acid (Aldrich, Milwauke , WI), 80.4 wt.% RCC-15C obtained without initiator (W.R. Grace, Atlanta, GA),

and 2 wt.% photoinitiator KB-1, and (b) 42.5 parts of BL036 liquid crystal mixture, with a solution viscosity of 210 cps (measured on a Brookfield viscometer using a #4 spindle operating at 60 rpm), was used. The die was configured with a 152 mm shim having a slot width of 88.9 cm, a 76 micron coating gap, and a 51 micron overbite. The coating was applied as a 88.9 cm wide strip of the uncured matrix on the ITO coated PET film at a substrate speed of 0.91 m/minute (3 feet/minute). During coating, a 3.7 mm Hg (2 inches water) vacuum was applied to the vacuum chamber. During lamination, an interference of 3.8 mm was used. The laminate construction was exposed to 330 mJ/cm^2 of UV light having an average intensity of 1.7 mW/cm^2.

The thickness of the cured coating was 21±0.6 microns. The PDLC device had on- and off-state transmissions of 74% and 2.7%, respectively, and a haze of 4.5%.

Example 11

A PDLC device was prepared as described in Example 7 except that a fluid containing (a) 45 parts of a mixture consisting of 2.5 wt.% KB-1 photoinitiator, 20.0 wt.% 9460 allyl aliphatic urethane (Monomer Polymer & Dajac, Trevose, PA), 35.0 wt.% isooctyl acrylate, 7.5 wt.% acrylic acid, 20 wt.% 2-phenoxyethyl acrylate, and 15.0 wt.% Uralac 3004-102, and (b) 55 parts of BL036 liquid crystal mixture, with a solution viscosity of 64 cps (measured on a Brookfield viscometer using a #3 spindle operating at 60 rpm), was used. The die was configured with a 152 micron shim having a slot width of 88.9 cm, an overbite of 30 microns and the coating applied to the ITO coated PET substrate at a rate of 3 m/min. at 20°C with a vacuum of 2.8 mm Hg applied to the vacuum chamber. An air pressur of 3.4 bar was maintain d to the lamination bar which was adjusted to provide an interference of

3.8 mm. The laminate construction was exposed to 303 mJ/cm^2 of UV light having an average intensity of 1.6 mW/cm^2.

The cured coating thickness was 17.4±0.6 microns. The PDLC device had on- and off-state transmissions of 70.0% and 0.8%, respectively, and a haze of 8.6%.

Example 12

An adhesive composition was prepared as described in Example 5 except that the prepolymer syrup was prepared from a solution containing 90 wt.% isooctyl acrylate, 10 wt.% acrylic acid, and 0.04 wt.% KB-1 photoinitiator that had been advanced to a viscosity of 430 cps (measured on a Brookfield viscometer using a # 4 spindle operating at 60 rpm) and to which an additional 0.1 wt.% KB-1 had been added was used as the coating fluid. The die was configured with a 0.25 mm shim, an overbite of 76 microns and a coating gap of 76 microns. The polymer syrup was cured in a N_2 atmosphere without a second substrate being applied to the coating by exposure to UV lights having an average intensity of 1.2 mW/cm^2 to produce a pressure sensitive adhesive having a thickness of 21.5±0.5 microns.

WHAT IS CLAIMED IS:

1. A method of coating the surface of a substrate with an essentially solvent-free polymerizable fluid comprising passing said fluid through a die onto the surface of said substrate as said substrate moves relative to said die,

said die comprising a channel adapted to receive said fluid and an adjustable width slot in communication with said channel, through which said fluid is passed, formed between a downstream bar and an upstream bar, said downstream bar having a die lip formed as a sharp edge and said upstream bar having a die lip formed as a land in a shape corresponding substantially to the shape of said substrate in the immediate area of coating fluid application to said substrate.

2. The method of claim 1 further comprising adjusting the rate at which said fluid passes through said die and rate at which said substrate moves relative to said die to produce a substantially uniform caliper coating on said substrate.

3. The method of claim 1 comprising configuring said sharp edge and said land such that said sharp edge is displaced towards the surface of said substrate relative to said land.

4. The method of claim 1 comprising providing a substantially straight, sharp edge in which the straightness of said edge measured along a distance of about 25 cm at any point along said sharp edge does not vary by more than about 2.5 microns.

5. The method of claim 1 comprising providing said land in the form of a curved land.

6. The method of claim 1 comprising providing said die with a convergence ranging from about 0° to about 2.29°.

7. The method of claim 1 comprising providing said substrate in the form of a web.

8. The method of claim 1 comprising providing a polymerizable fluid having a viscosity of at least 10 cps.

9. A method of coating the surface of a substrate with an essentially solvent-free polymerizable fluid comprising the steps of:

(a) passing said fluid through a die onto the surface of said substrate as said substrate moves relative to said die,

said die comprising a channel adapted to receive said fluid and an adjustable width slot in communication with said channel through which said fluid is passed formed between a substantially straight, sharp edge located on the downstream side of said substrate and a land located on the upstream side of said substrate; and

(b) adjusting the rate at which said fluid passes through said die and rate at which said substrate moves relative to said die to produce a substantially uniform caliper coating on said substrate.

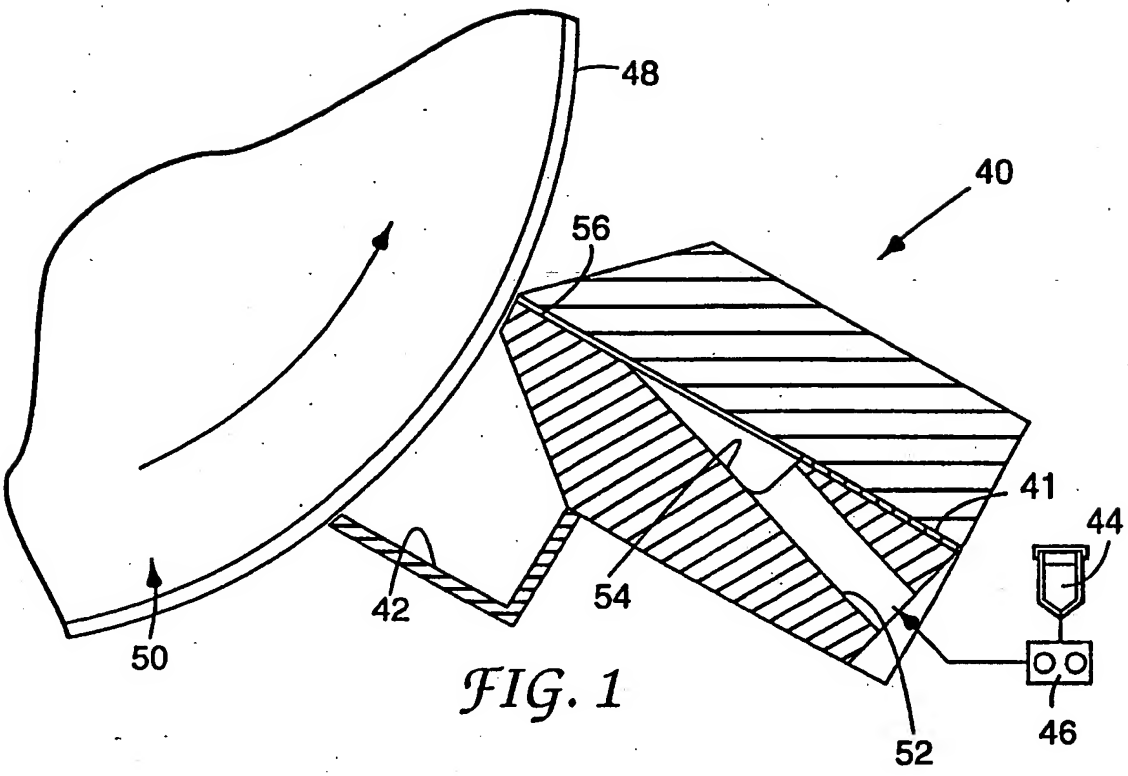


FIG. 1

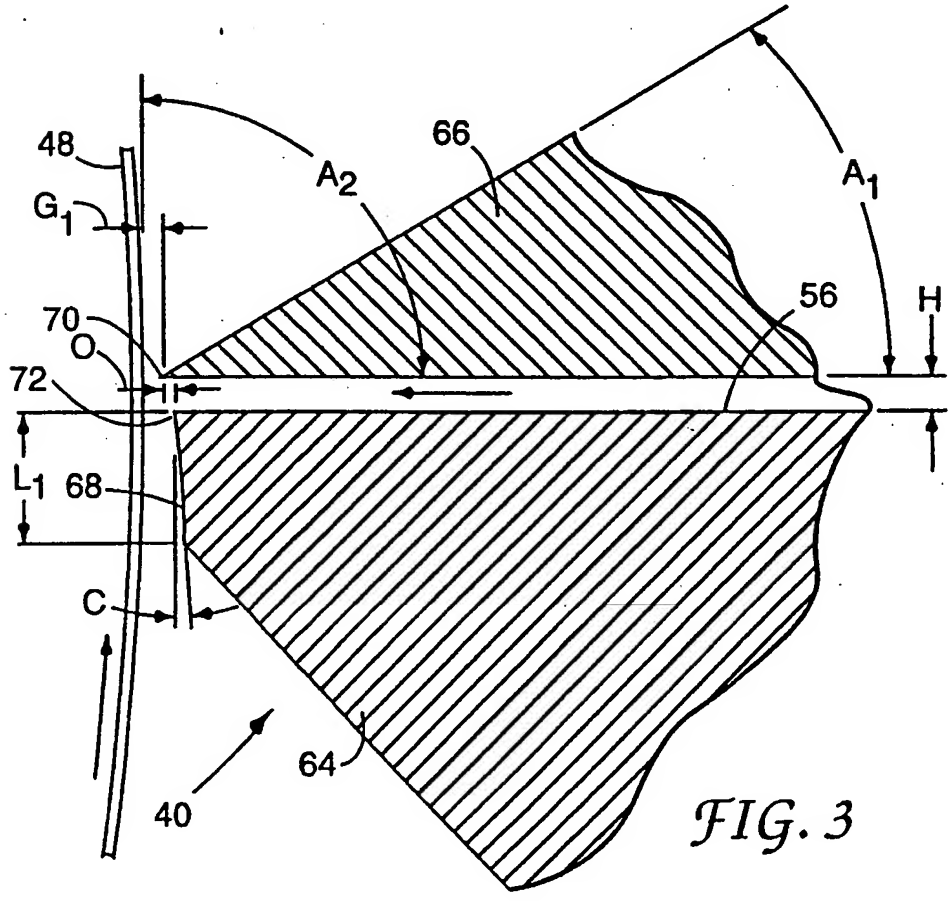


FIG. 3

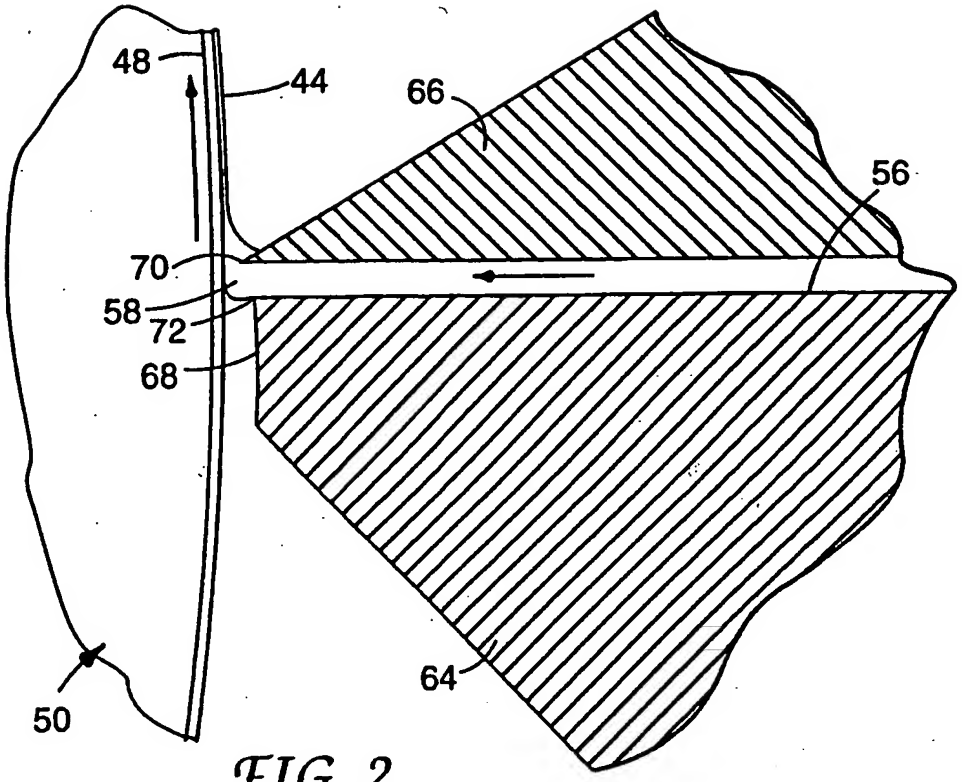


FIG. 2

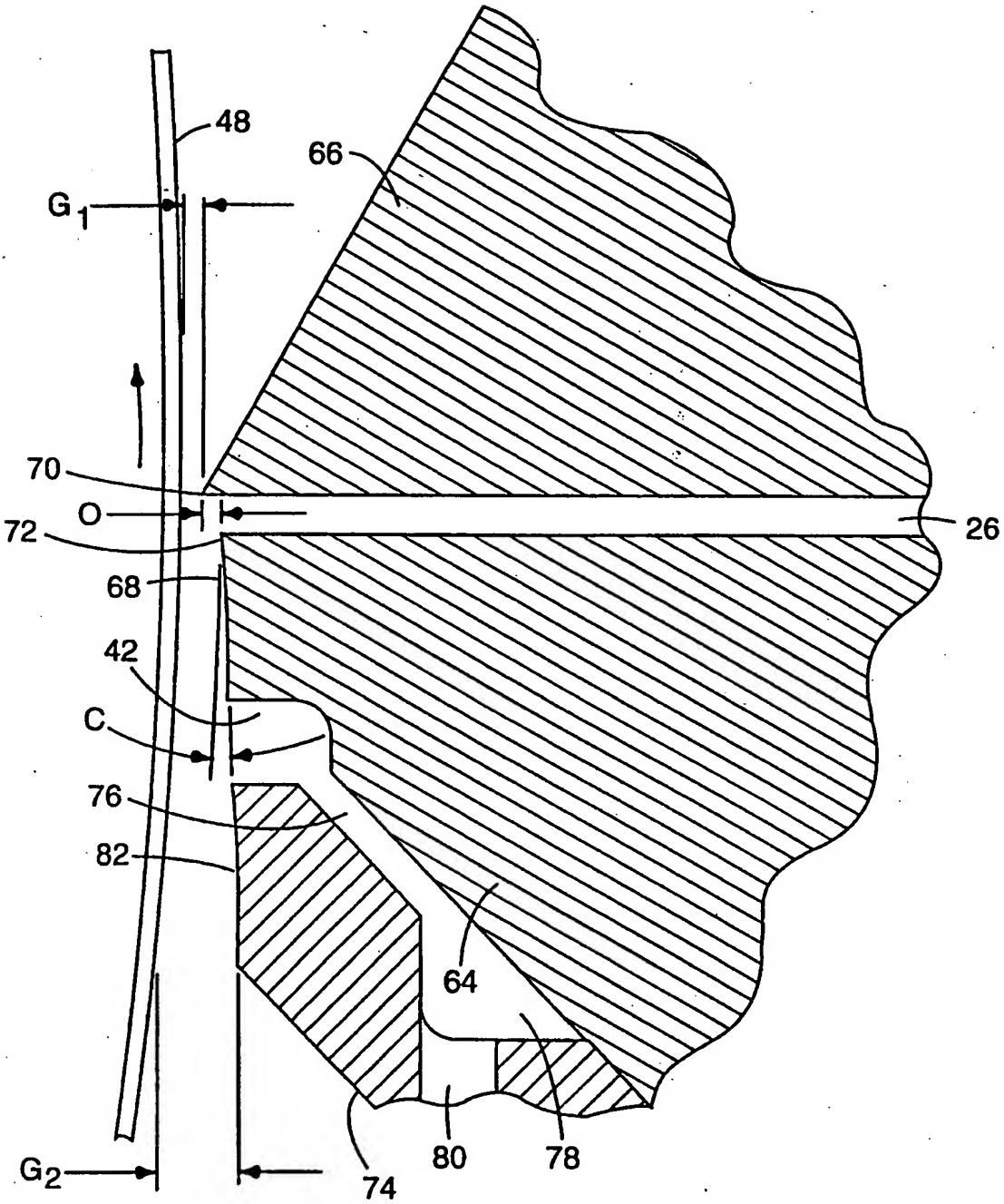
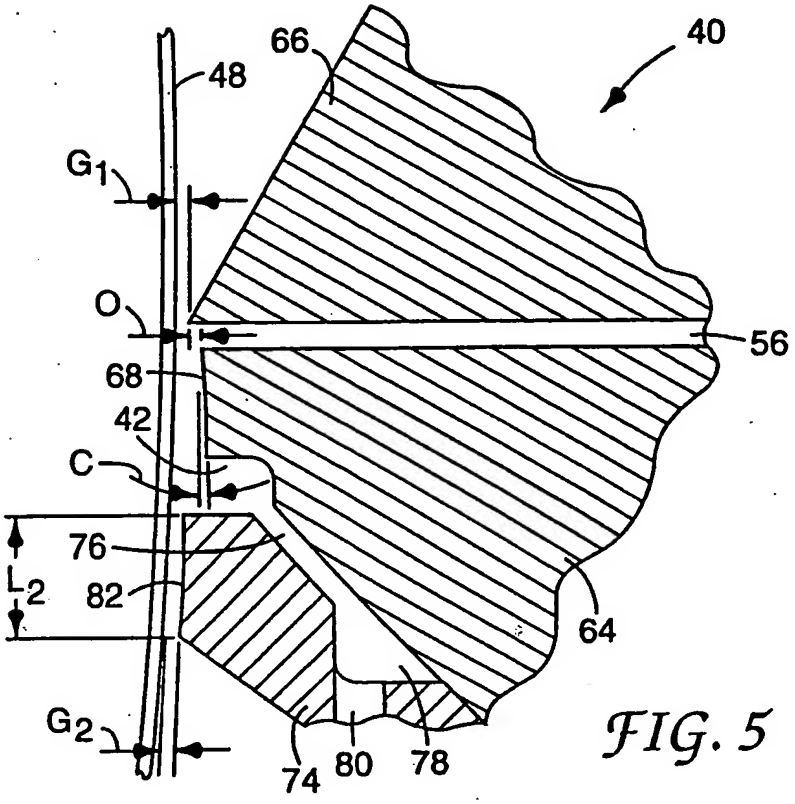


FIG. 4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L2
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G2
74
80
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FIG. 5

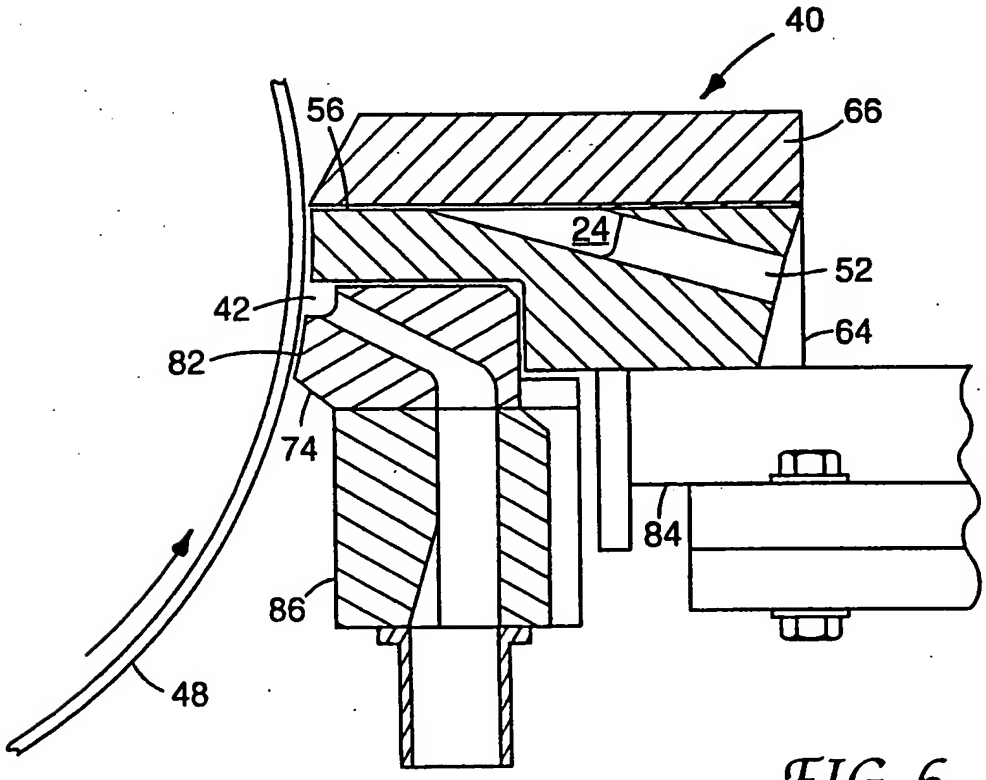
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FIG. 6

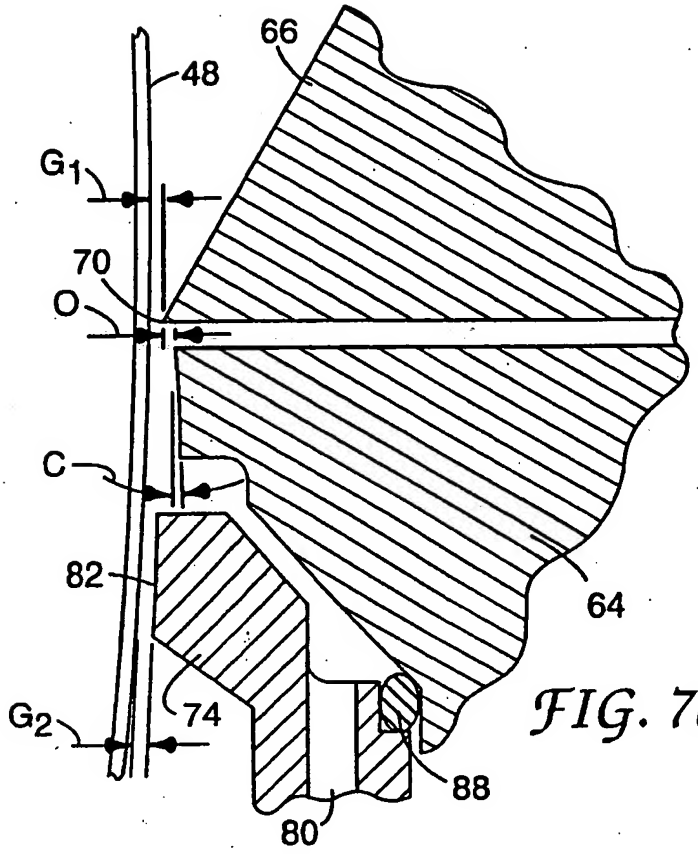
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FIG. 7a

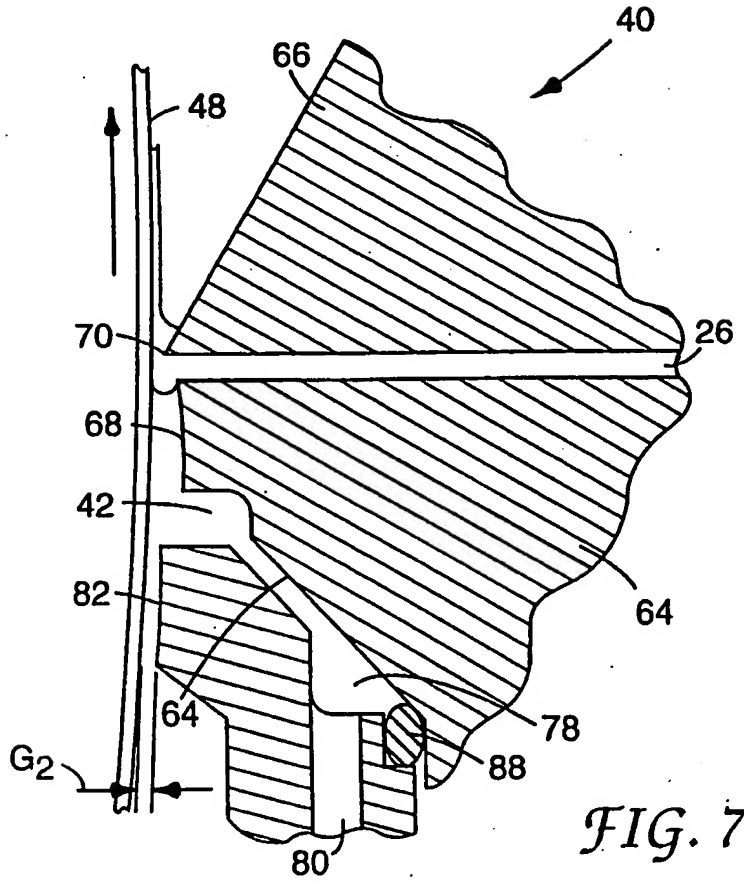
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FIG. 7b

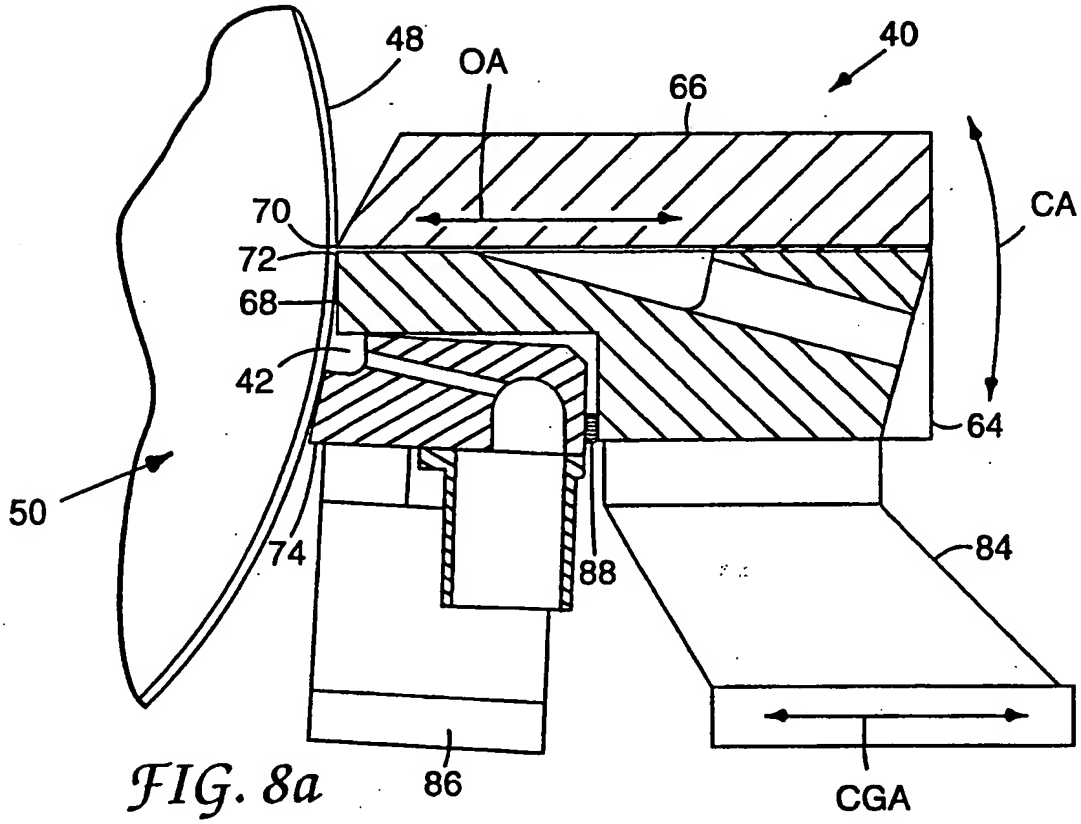

FIG. 8a

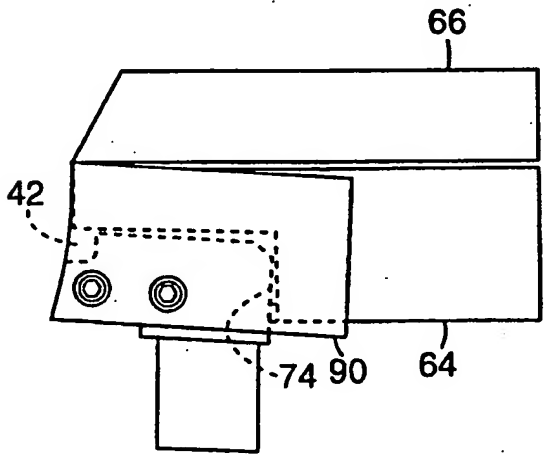

FIG. 8b

INTERNATIONAL SEARCH REPORT

Inter nal Application No
PCT/US 95/04466

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 B05C5/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 B05C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US,A,4 445 458 (E.I. DU PONT DE NEMOURS AND CO) 1 May 1984 see the whole document	1-3,7,9
A	DE,A,43 36 365 (VALMET PAPER MACHINERY INC) 28 April 1994 see the whole document	1

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
24 August 1995	06.10.95

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax (+31-70) 340-3016	Klinger, T

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Interr ual Application No
PCT/US 95/04466

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
US-A-4445458	01-05-84	NONE		
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		CA-A-	2108841	27-04-94
		FR-A-	2697176	29-04-94
		GB-A,B	2272390	18-05-94
		JP-A-	6198242	19-07-94
		SE-A-	9303494	27-04-94